

# Infrared Spectra of Plastics and Resins

## Part 3 - Related Polymeric Materials (Elastomers)

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Previous Reports in this Series

"Infrared Spectra of Plastics and Resins," R. E. Kagarise and  
L. A. Weinberger, NRL Report 4369, May 26, 1954

"Part 2 - Materials Developed Since 1954," Suzanne S. Stimler  
and R. E. Kagarise, NRL Report 6392, May 23, 1966

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## ABSTRACT

Infrared spectra of thin films of 47 elastomers have been obtained in the region from 400 to 4000  $\text{cm}^{-1}$ . The elastomers studied were uncured natural and synthetic rubbers, chosen as representative of the various classes of rubbers.

The identification chart presented in Parts 1 and 2 of this series of reports has been expanded to include the elastomers studied here, in addition to the plastics used in that work. The use of this expanded chart and the reference spectra should make it possible to determine from the infrared spectrum of an unknown polymer its class or type.

## PROBLEM STATUS

This is an interim report; work on this problem is continuing.

## AUTHORIZATION

NRL Problem C07-01  
Project RR 001-01-43-4803

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## INFRARED SPECTRA OF PLASTICS AND RESINS

### Part 3 — Related Polymeric Materials (Elastomers)

#### INTRODUCTION

Rubber technology has become increasingly complex over the past 25 years. Beginning with the development of the first synthetic rubber in 1930, an industry has grown to meet the demands for new synthetic elastomers having highly specific properties. As the number of elastomers grows, so must the methods of identifying these products grow. Infrared spectroscopy has been applied to this problem at this Laboratory (1) and elsewhere (2-8). The present work is intended to extend these previous investigations by including elastomers representative of all the major types available today.

Dinsmore and Smith (1) found that nearly identical spectra were obtained from pure gum elastomers and the corresponding cured rubbers after additives had been removed by extraction. This indicated that the curing process changed relatively few recurring units of the polymer and thus had little effect on the spectrum. In the present work, therefore, the elastomers were studied in their simplest form, the raw or uncured state. The presentation of the spectra of the raw elastomers, without the presence of additives used in the curing process, provides a convenient reference for comparison with spectra obtained from unknown elastomers.

#### EXPERIMENTAL METHOD

The spectra in this study were obtained by using a Beckman IR-12 grating spectrophotometer in the region from 400 to 4000  $\text{cm}^{-1}$ . The normal operating parameters for the spectrophotometer were: scanning speed, 80  $\text{cm}^{-1}/\text{min}$ ; gain, 3%; time constant, 2 sec; and single-beam/double-beam energy ratio, 1. In nearly all cases the elastomers were studied as films cast from solution onto potassium bromide plates. The only exception was the Thiokol A sample, which, because of its insolubility in common laboratory solvents, was prepared by melting the rubber between salt plates.

The solvents used in the preparation of films were benzene, acetone, methyl ethyl ketone, ethyl acetate, and 1,2-dichloroethane. Evaporation at room temperature was usually effective in removing the solvent. However, with methyl ethyl ketone, and in some cases benzene, excess solvent remained trapped after evaporation, and it was necessary to heat the film in vacuum at about 60°C for two hours in order to remove the solvent.

#### RESULTS

Table 1 gives the commercial name, chemical name or type, manufacturer, and the elastomer class for the elastomers studied. The solvents used in the preparation of the samples and the spectrum numbers for the elastomers are also shown. The collection of spectra at the back of the report is indexed by an alphabetical listing of the rubbers according to rubber class. The commercial and chemical names and the manufacturer are given, where appropriate, on each spectrum in addition to the general structure of the elastomer studied.

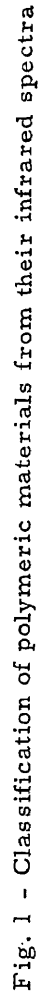
Table 1  
Elastomer Data

Elastomer Class	Commercial Name	Chemical Name or Type	Manufacturer	Spectrum No. <sup>a</sup>	Solvent
Isoprene rubber	Natural rubber (pale crepe)	Polyisoprene	--	1	Benzene
	Natural rubber (smoked sheet)	Polyisoprene	--	2	Benzene
	Deproteinized rubber	Polyisoprene	--	1†	Benzene
	IR-309	Polyisoprene	Shell	3†	Benzene
	IR-350	Polyisoprene	Shell	3†	Benzene
	IR-501	Polyisoprene	Shell	3	Benzene
Polysulfide rubber	Thiokol A	Organic polysulfide	Thiokol	4	Melted between salt plates
	Thiokol FA	Organic polysulfide	Thiokol	5	1,2-Dichloroethane
	Thiokol ST	Organic polysulfide	Thiokol	5†	1,2-Dichloroethane
	Thiokol LP-3	Organic polysulfide	Thiokol	5†	1,2-Dichloroethane
Chloroprene rubber	Neoprene FB	Polychloroprene	du Pont	6	Benzene
	Neoprene FC	Polychloroprene	du Pont	7a amorphous 7b crystalline	Benzene
	Neoprene GN-A	Polychloroprene	du Pont	6†	Benzene
	Neoprene WB	Polychloroprene	du Pont	6†	Benzene
	Neoprene WRT	Polychloroprene	du Pont	6†	Benzene
Butadiene rubber and copolymers	Ameripol CB 220	"cis"-1,4-Polybutadiene	Goodrich-Gulf	8	Benzene
	Budene 501	"cis"-1,4-Polybutadiene	Goodyear	8†	Benzene
	Hycar 1011	Butadiene-acrylonitrile copolymer	Goodrich	9†	Benzene
	Hycar 1014	Butadiene-acrylonitrile copolymer	Goodrich	9	Benzene
	Hycar 1042 x 100	Butadiene-acrylonitrile copolymer	Goodrich	9†	Benzene
	Paracril AJ	Butadiene-acrylonitrile copolymer	Naugatuck	9†	Benzene
	Philprene 1500	Butadiene-styrene copolymer	Phillips	10	Benzene
Isobutylene-isoprene rubber	Butyl 035	Isobutylene-isoprene copolymer	Enjay	11	Benzene
	Hycar 2202	Isobutylene-isoprene copolymer (with 2 to 3 wt-% bromine)	Goodrich	12	Benzene
	Butyl HT 10-66	Isobutylene-isoprene copolymer (with 1.1 to 1.3 wt-% chlorine)	Enjay	11†	Benzene
Silicone rubber	Silastic 400 SE-33	Poly(dimethylsiloxane)	Dow	13	Benzene
		Mixed methyl vinyl silicone, low vinyl	G.E.	13†	Benzene
	Silastic 410	Mixed methyl vinyl silicone, high vinyl	Dow	14	Benzene
	Silastic 440	Mixed methyl phenyl vinyl silicone	Dow	15	Benzene
Acrylic rubber	Acrylon BA-12	Acrylic ester-acrylonitrile copolymer	Borden	16	Benzene
	Hycar 1072	Carboxyl-modified butadiene-acrylonitrile copolymer	Goodrich	17	Benzene
	Thiacril 76	Ethyl acrylate base elastomer	Thiokol	18	Benzene
	Hycar 4021	Acrylic ester polymer	Goodrich	18†	Benzene
Chlorosulfonated polyethylene	Hypalon 20	Chlorosulfonated polyethylene	du Pont	19†	Benzene
	Hypalon 30	Chlorosulfonated polyethylene	du Pont	19†	Methyl ethyl ketone
	Hypalon 40	Chlorosulfonated polyethylene	du Pont	19a without talc 19b with talc	Benzene
Urethane elastomer	Elastothane 455	Polyurethane	Thiokol	20	Benzene
Fluorinated rubber	Silastic LS-63	Fluorosilicone	Dow	21	Acetone
	Viton A-HV	Perfluoropropylene-vinylidene fluoride copolymer	du Pont	22	Methyl ethyl ketone
	Viton B	Perfluoropropylene-vinylidene fluoride copolymer	du Pont	23	Methyl ethyl ketone
	Kel-F 3700	Chlorotrifluoroethylene-vinylidene fluoride copolymer	3M	24†	Ethyl acetate
	Kel-F 5500	Chlorotrifluoroethylene-vinylidene fluoride copolymer	3M	24	Ethyl acetate
Ethylene-propylene rubber	EPR 404	Ethylene-propylene copolymer	Enjay	25†	Benzene
	EPT 3509	Ethylene-propylene-diene terpolymer	Enjay	25	Benzene
	Nordel 1070	Ethylene-propylene-diene terpolymer	du Pont	25†	Benzene
Epichlorohydrin	Hydrin 100	Polyepichlorohydrin	Goodrich	26	Benzene
	Hydrin 200	Epichlorohydrin-ethylene oxide copolymer	Goodrich	27	Benzene

<sup>a</sup>These numbered spectra are given at the end of the report.

†The spectrum indicated is not the spectrum of the elastomer named, but the two are identical, except for intensity variations.

Figure 1 diagrams the steps of a procedure that can be used in the classification of unknown polymeric materials from their infrared spectra. It is an extension and slight modification of the chart presented in the previous report in this series (9), the elastomers studied here having been added to that chart. This chart should serve merely as a guide, with final identification resting on the comparison of the unknown and reference spectra.



## DISCUSSION

Since spectra of many of the elastomers studied have been discussed elsewhere, detailed discussions will be omitted here. Certain interesting spectral features will be pointed out, however, and some characteristics of the rubber classes (10,11) will be noted.

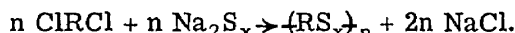
## Isoprene Rubbers

Natural rubber is a "cis"-1,4 polymer of isoprene found in the latex of rubber trees. The latex is a dispersion of rubber in water in which the dispersing agent is a natural protein material. There are also variable small amounts of nonrubber substances present in the latex. Different types of natural rubber are obtained, depending on the processing given the latex. The three types used in this study (pale crepe, smoked sheet, and deproteinized rubber) gave nearly identical spectra (spectra 1 and 2) which, for the most part, may be interpreted in terms of group vibrations based on the chemical structure (1,3,4,6). Weak absorption at 1635 and 3300  $\text{cm}^{-1}$  by smoked sheet (spectrum 2) may be due to residual water in this rubber. At present no explanation can be given for the band at 1540  $\text{cm}^{-1}$ .

Synthetic isoprene rubbers were also studied. These are produced so as to have stereospecific "cis" structure comparable to that of natural rubber. The spectra of these samples duplicated that of the natural rubber, except for intensity differences of some bands and an additional weak absorption at 1645  $\text{cm}^{-1}$ ; a typical example is given for isoprene rubber IR-501 in spectrum 3. The increased absorption at 888 and 1645  $\text{cm}^{-1}$  in spectrum 3 over that in spectrum 1 is probably due to the presence of some 3,4-polyisoprene in the IR-501 polymer (3).

## Polysulfide Rubbers

The first synthetic rubber made in this country was Thiokol A. This and other polysulfide rubbers are formed by reactions between sodium polysulfides and organic dihalides. The general reaction may be represented by



Thiokol A (spectrum 4) is made from  $\text{ClCH}_2\text{CH}_2\text{Cl}$  and sodium polysulfide of composition  $\text{Na}_2\text{S}_4$ , Thiokol ST is made from  $\text{ClCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{Cl}$  and  $\text{Na}_2\text{S}_{2.25}$ , and Thiokol FA (spectrum 5) is made from  $\text{ClCH}_2\text{CH}_2\text{Cl}$ ,  $\text{ClCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{Cl}$ , and  $\text{Na}_2\text{S}_{1.8}$  (10). Agents are also added during the polymerization of Thiokol ST to produce some crosslinking. Liquid polymers (such as Thiokol LP-3) are produced when disulfide links in the high-molecular-weight polymers are reduced. Dichlorodiethylformal is the halide used in making the liquid polymers, and some crosslinking is introduced through the use of 0.5 to 2 mole-% of a trifunctional compound, a trichloropropane. All of the polysulfide rubbers have excellent solvent resistance.

Because of the complexity of the spectra of Thiokols, band assignments cannot be readily made. Very few differences (variations in some band intensities) are noted between the spectra of Thiokol FA and Thiokol ST. The higher proportion of sulfur in Thiokol A over that in Thiokol FA and ST results in increased absorption in the S-S and C-S stretching regions (400 to 500 and 600 to 700  $\text{cm}^{-1}$ ; respectively) in the Thiokol A spectrum (spectrum 4). At present, no explanation can be given for the other differences in the spectra of Thiokol A, FA, and ST.

## Chloroprene Rubbers

Neoprene, a polymer of chloroprene, is a good general-purpose rubber. Its relatively high cost of manufacture, however, has limited its use to those applications that require its unique properties, such as resistance to oils, chemicals, air, light, heat, and flame.



The infrared spectrum of neoprene has been discussed previously (1,3,4). All five neoprenes studied exhibit similar spectra differing only in the relative intensity of certain bands in the 400 to 1700  $\text{cm}^{-1}$  region. The weak band at 1585  $\text{cm}^{-1}$  present in the spectra of Neoprene FB and GN-A may be due to additives in these elastomers (spectrum 6).

When neoprene crystallizes, prominent bands appear in the spectrum at 782 and 952  $\text{cm}^{-1}$ . From the ratio of the absorbance of these bands to that of the CH absorption at 2940  $\text{cm}^{-1}$ , the degree of crystallinity of neoprene may be determined (5). The infrared spectra of Neoprene FC films indicate that crystallization takes place as the film ages. Spectrum 7a is that of a freshly prepared film, while spectrum 7b is that of the film one day later. The bands at 782 and 952  $\text{cm}^{-1}$  are very apparent in the spectrum of the old film.

#### Butadiene Rubbers and Copolymers

Butadiene plays a major role in the synthetic rubber industry. Stereospecific polybutadienes have been developed which can be used in place of natural rubber, imparting improved resilience and low-temperature flexibility. Copolymerization with acrylonitrile produces a rubber resistant to oils, while use of styrene as the comonomer improves elongation and strength. Rubbers of these three types were included in this study.

Infrared spectra of some butadiene rubbers have been discussed in detail by other workers (1,3,4). The outstanding feature of the spectrum of "cis"-polybutadiene (spectrum 8) is the intense band at 730  $\text{cm}^{-1}$  arising from the out-of-plane CH bending mode of the  $-\text{CH}=\text{CH}-$  group. The position of this band and that of the C=C stretching mode at 1655  $\text{cm}^{-1}$  confirm that this polymer is mostly of the "cis" structure (3). The presence of bands at 970 and 1667  $\text{cm}^{-1}$  in spectra 9 and 10 shows the copolymers to contain butadiene of mostly "trans" structure. Both spectra show characteristic bands of the component monomers, acrylonitrile being identified by the sharp  $\text{C}\equiv\text{N}$  band at 2220  $\text{cm}^{-1}$  (spectrum 9) and styrene by aromatic bands at 700, 1495, and 1600  $\text{cm}^{-1}$  (spectrum 10). The several butadiene-acrylonitrile copolymers studied differed only in intensity variations of some bands, undoubtedly the result of differences in monomer ratios in the various copolymers.

#### Isobutylene-Isoprene Rubbers

The copolymerization of isobutylene with small amounts of isoprene gives a rubber-like product called Butyl rubber. Butyl rubbers have good resistance to chemicals and oxidation and have low air permeability. Hycar 2202, a brominated Butyl rubber, has good adhesion properties, while Butyl HT 10-66, a chlorinated Butyl rubber, has good high-temperature resistance.

Unmodified Butyl rubber, as well as chlorinated and brominated samples, were studied. All three types had similar spectra since the amount of Cl and Br (from 1 to 3 wt-%) in the halogenated rubbers is too small to give appreciable C-Cl or C-Br absorption. A small C-Br stretching vibration is detected at 450  $\text{cm}^{-1}$  in spectrum 12, but the spectrum of the chlorinated Butyl rubber is essentially the same as the unmodified rubber spectrum (spectrum 11). Weak absorption at 1720 and 1750  $\text{cm}^{-1}$  by the modified rubbers (spectrum 12) presumably arises from stabilizers or other additives.

#### Silicone Rubbers

The silicone rubbers are polysiloxanes having various hydrocarbon substituents. The three main types, according to pendant groups, are dimethyl-, methylvinyl-, and methylphenylsiloxane. The physical properties of the rubbers are relatively poor, but they have the widest useful temperature range ( $-150^{\circ}$  to  $450^{\circ}\text{F}$ ) of any of the elastomers.

Most of the bands in the silicone spectra may be explained in terms of the chemical structure given for these elastomers. The vinyl content of SE-33 was not sufficient to show in the spectrum, and the absorption of this rubber was identical to that of dimethylsiloxane (Silastic 400, spectrum 13). Silastic 410 (spectrum 14) has a higher vinyl content and does show weak absorption at 1600 and 3060  $\text{cm}^{-1}$ , which distinguishes it from Silastic 400. Absorptions from CH and  $\text{CH}_2$  bending modes of the  $-\text{CH}=\text{CH}_2$  groups in Silastic 410 are probably hidden by the strong Si-O-Si absorption in the 980 to 1150  $\text{cm}^{-1}$  region.

### Acrylic Rubbers

Acrylic rubbers are polymers or copolymers of acrylic esters. The absence of residual unsaturation in the molecules makes them resistant to degradation by sulfur and oxygen and thus able to perform well in the presence of sulfur-modified oils at temperatures above 350°F. Spectra 16 to 18 are absorption curves for rubbers of this class, each curve showing bands characteristic of the functional groups of the component monomers.

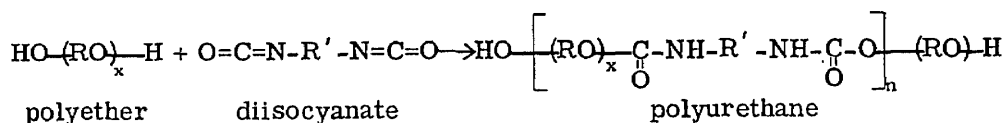
### Chlorosulfonated Polyethylene

Hypalon is polyethylene modified by substitution of chlorine and sulfonyl chloride groups on the chain, with a ratio of approximately one Cl atom for each seven carbon atoms and one  $\text{SO}_2\text{Cl}$  group for every 90 carbon atoms. Chlorine serves to destroy crystallinity, and  $\text{SO}_2\text{Cl}$  provides sites of reactivity for vulcanization.

The infrared spectrum of Hypalon has been discussed elsewhere (6,7). Spectrum 19 is representative of Hypalons 20, 30, and 40, all of which have nearly identical absorption curves. Increased absorption at 455, 470, 675, and 1025  $\text{cm}^{-1}$  was obtained if the dusting agent used on the rubbers (presumably talc) was not removed. Spectrum 19a is the absorption of Hypalon 40 free of talc, and spectrum 19b is for the elastomer containing suspended talc.

### Urethane Elastomers

Polyurethanes can result from the reaction of diisocyanates with reactive groups in polyethers or polyesters. A typical reaction is



where R and R' are alkyl groups. A variety of polyethers, polyesters, diisocyanates, and crosslinking agents are used in the preparation of urethane elastomers.

The infrared absorption of Elastothane 455, a representative polyurethane elastomer, is given in spectrum 20. Most of the bands may be explained in terms of NH,  $\text{CH}_2$ ,  $\text{CH}_3$ , C=O, and C-O-C groups.

### Fluorinated Rubbers

Several types of elastomers can be grouped under the heading fluorinated rubbers, their common characteristic being their fluorine content. It is this fluorine content which makes these compounds so valuable for their solvent resistance at high temperatures. The types of fluoroelastomers studied include a fluorosilicone (spectrum 21), perfluoropropylene-vinylidene fluoride copolymers (spectra 22 and 23), and chlorotrifluoroethylene-vinylidene fluoride copolymers (spectrum 24). The spectra of all these rubbers show strong C-F absorption in the 1000 to 1300  $\text{cm}^{-1}$  region and  $\text{CF}_2$  absorption in the 500 to 700  $\text{cm}^{-1}$  region. The other regions of the spectra are characteristic of the other structural units in the individual elastomers.

### Ethylene-Propylene Rubbers

The absorption curves for the ethylene-propylene rubbers are very simple, since the materials contain only  $-\text{CH}_2-$  and  $-\text{CH}_3$  groups and, in the case of the terpolymers, an occasional  $-\text{CH}=\text{CH}-$  group. The infrared absorptions for these rubbers were all similar, and a representative curve is given in spectrum 25.

### Epichlorohydrin Elastomers

The epichlorohydrin elastomers are recently developed specialty rubbers resistant to solvents, oils, ozone, and heat. Their spectra (spectra 26 and 27) show characteristic absorptions for C-O-C, C-Cl, and CH vibrations. The different amounts of chlorine in Hydrin 100 and 200 are evident from the spectra. The intensity ratio of the C-Cl band at  $750\text{ cm}^{-1}$  to the C-H band at  $2880\text{ cm}^{-1}$  is higher in the more highly chlorinated polymer, Hydrin 100.

### CONCLUSION

Obviously, not all of the elastomers available today have been investigated in this work, but an attempt was made to include at least one material from every major rubber class and subclass. Such a representative sampling is entirely satisfactory for the limited identification that is possible for members of many rubber classes. The only difference between elastomers within a class is often the small variation in proportions of component monomers, which is detectable in the infrared spectrum as a slight variation in the intensities of certain bands.

The addition of the elastomers studied to the classification chart presented in Part 2 of this series makes the chart helpful in the identification of a large variety of unknown polymeric materials.

### ACKNOWLEDGMENTS

The assistance of Messrs. E. J. Kohn and Ralph C. Taylor of the Chemistry Division in obtaining samples and providing information about the elastomers is gratefully acknowledged.

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Deproteinized rubber*	1
IR-309*	3
IR-350*	3
IR-501	3
Natural rubber (pale crepe)	1
Natural rubber (smoked sheet)	2

**IX. Isobutylene-isoprene rubbers**

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Thiokol ST*	5

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Silastic 400	13
Silastic 410	14
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**XII. Urethane elastomers**

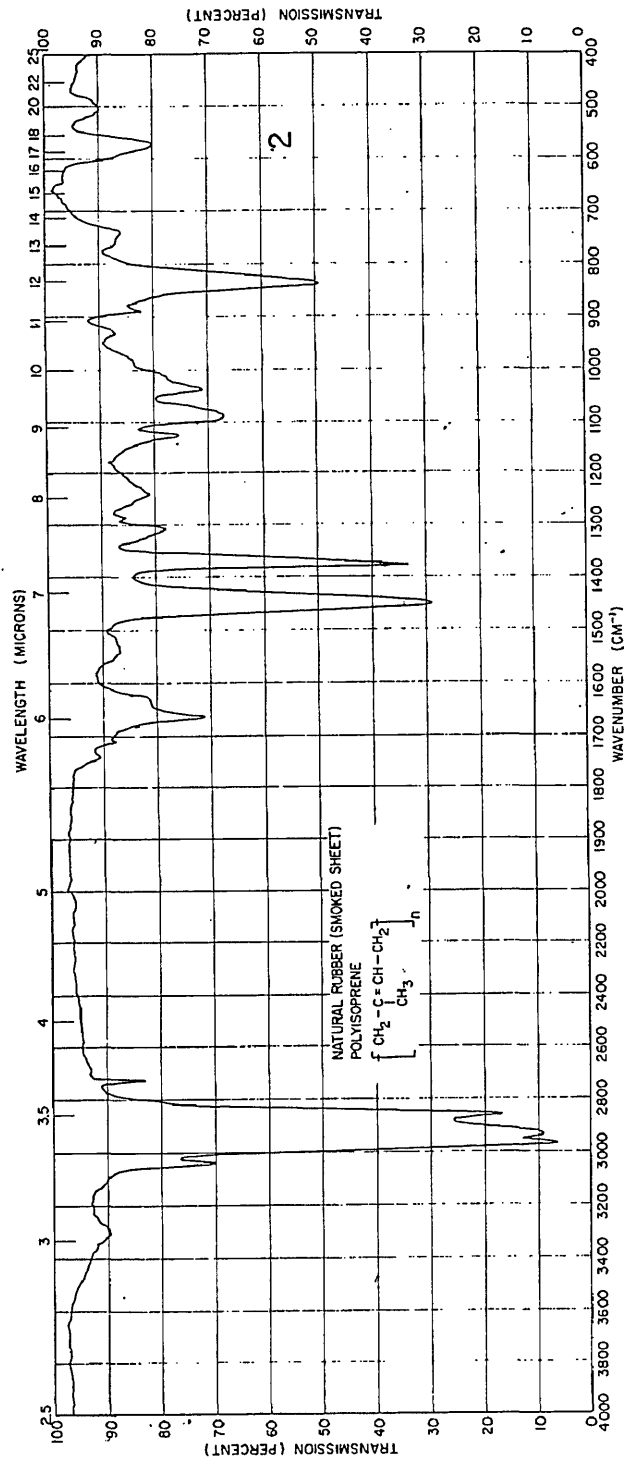
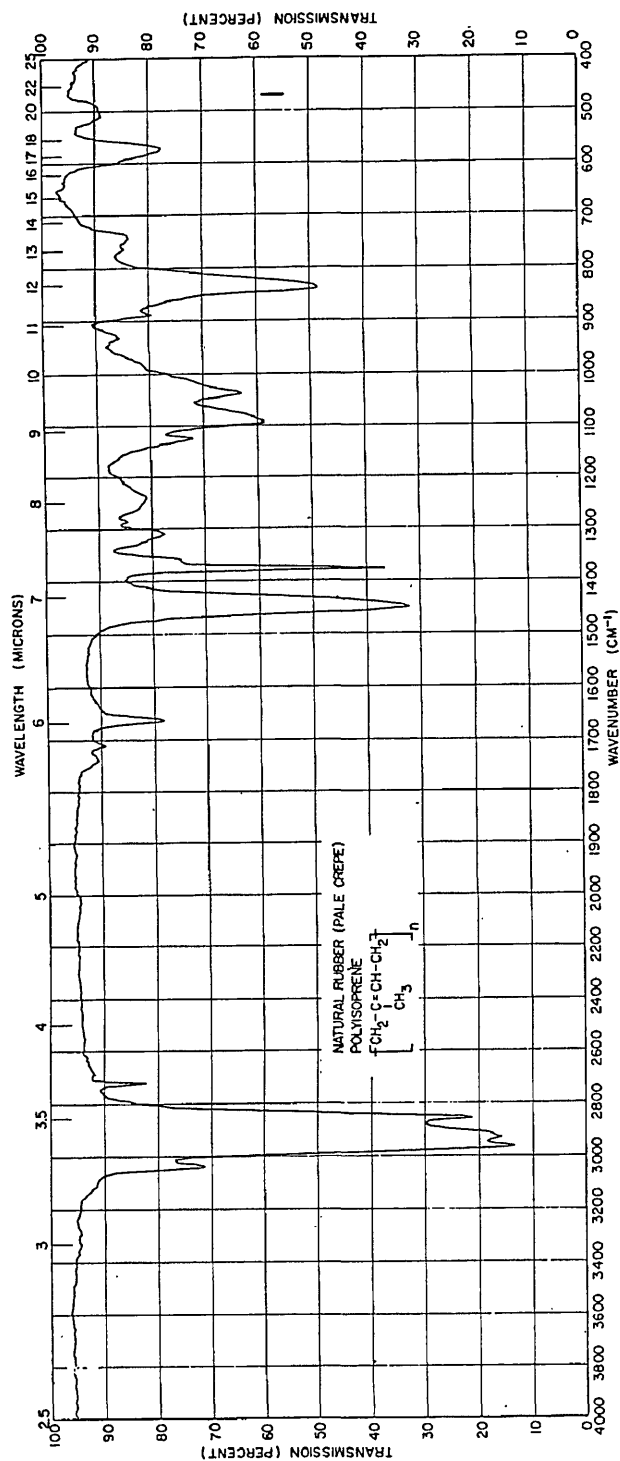
Elastothane 455	20
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\*The spectrum for this rubber is not presented, but the number given is for an identical spectrum, except for intensity variations.

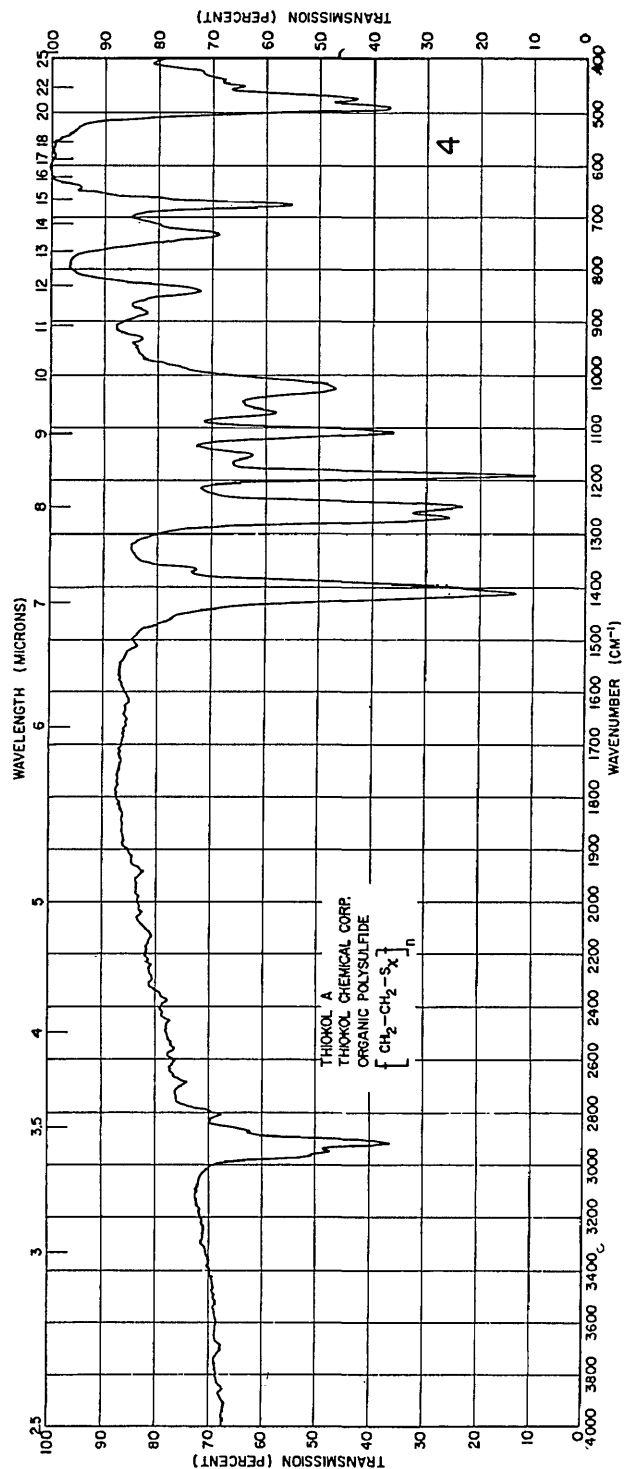
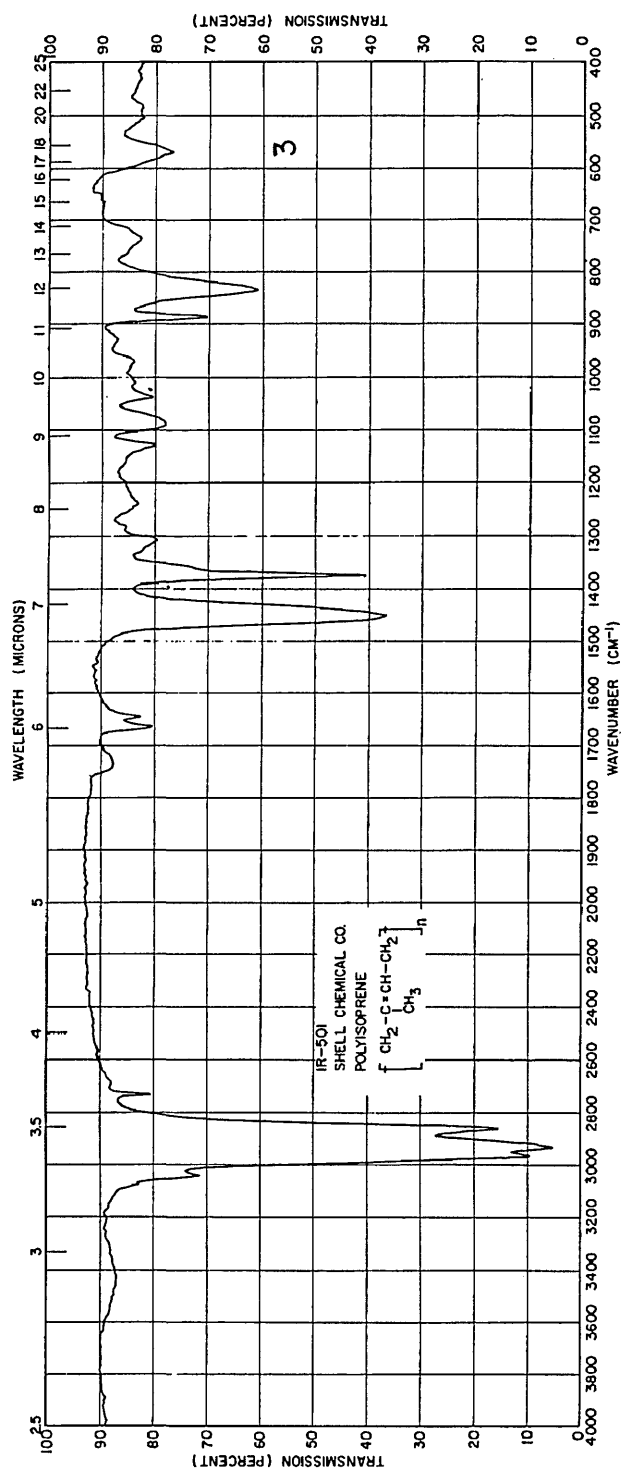
†Curve a is for freshly prepared film; curve b is for day-old film.

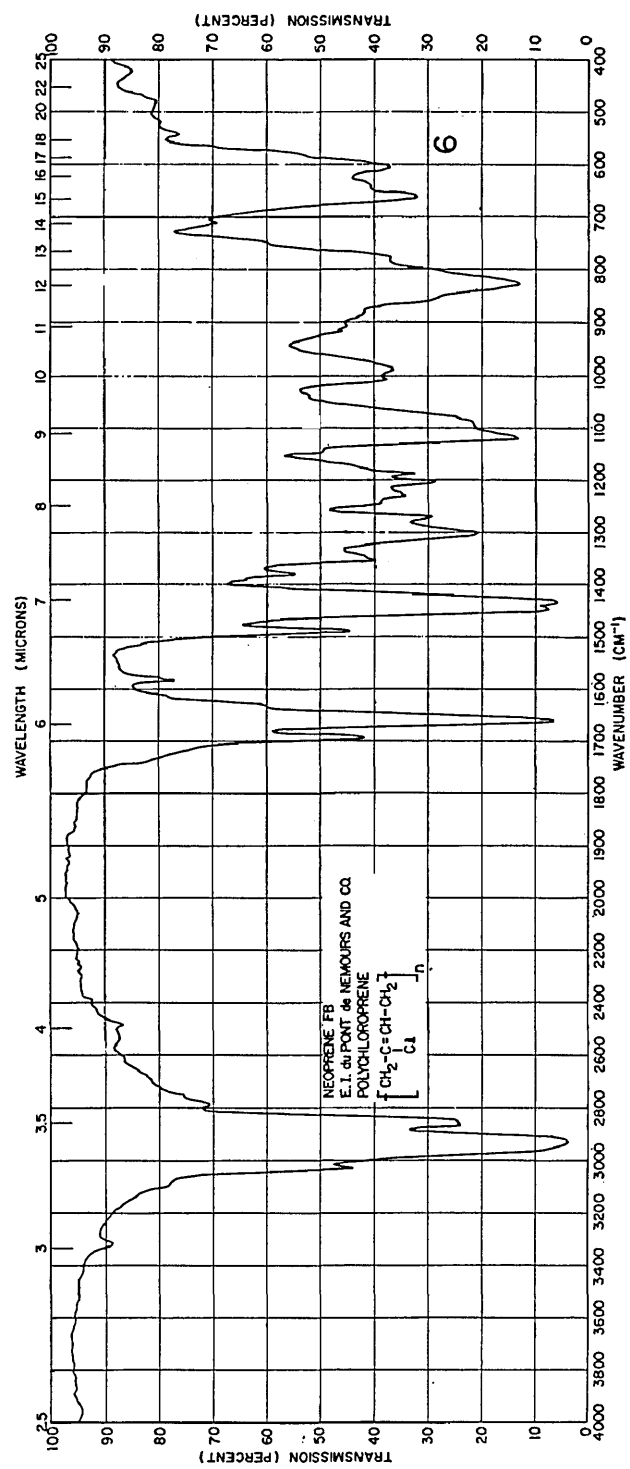
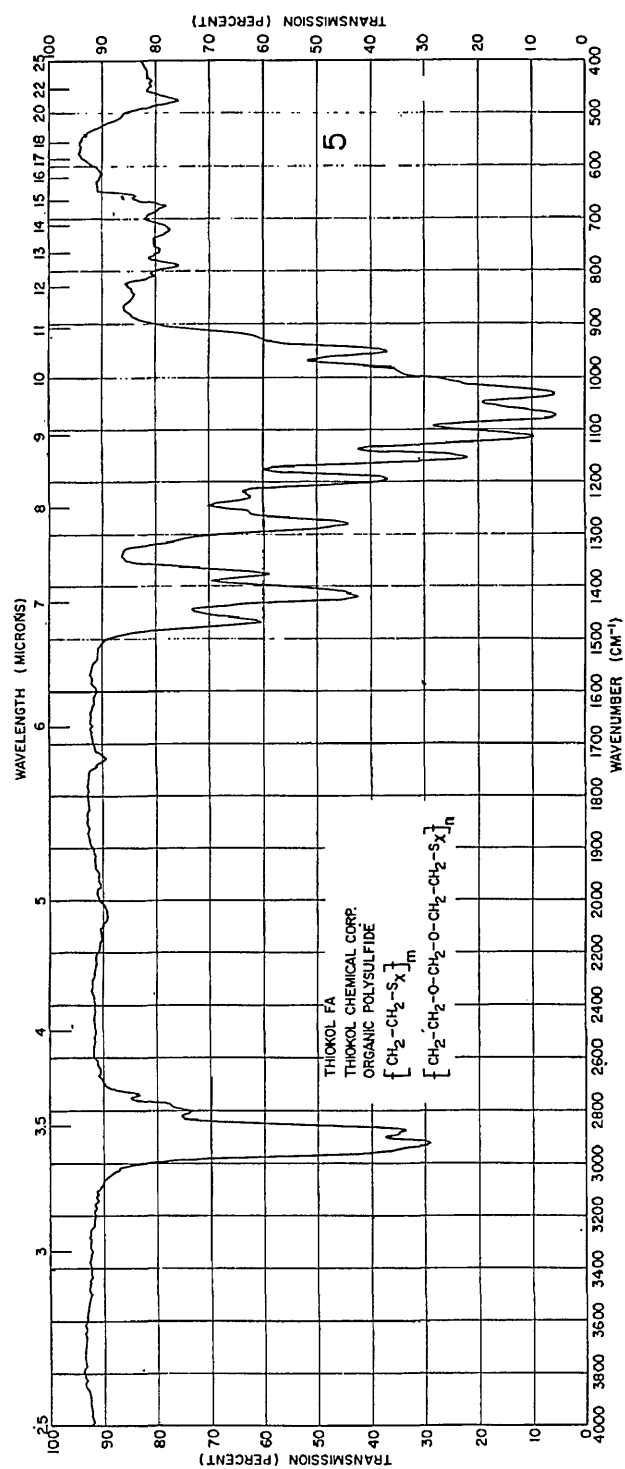
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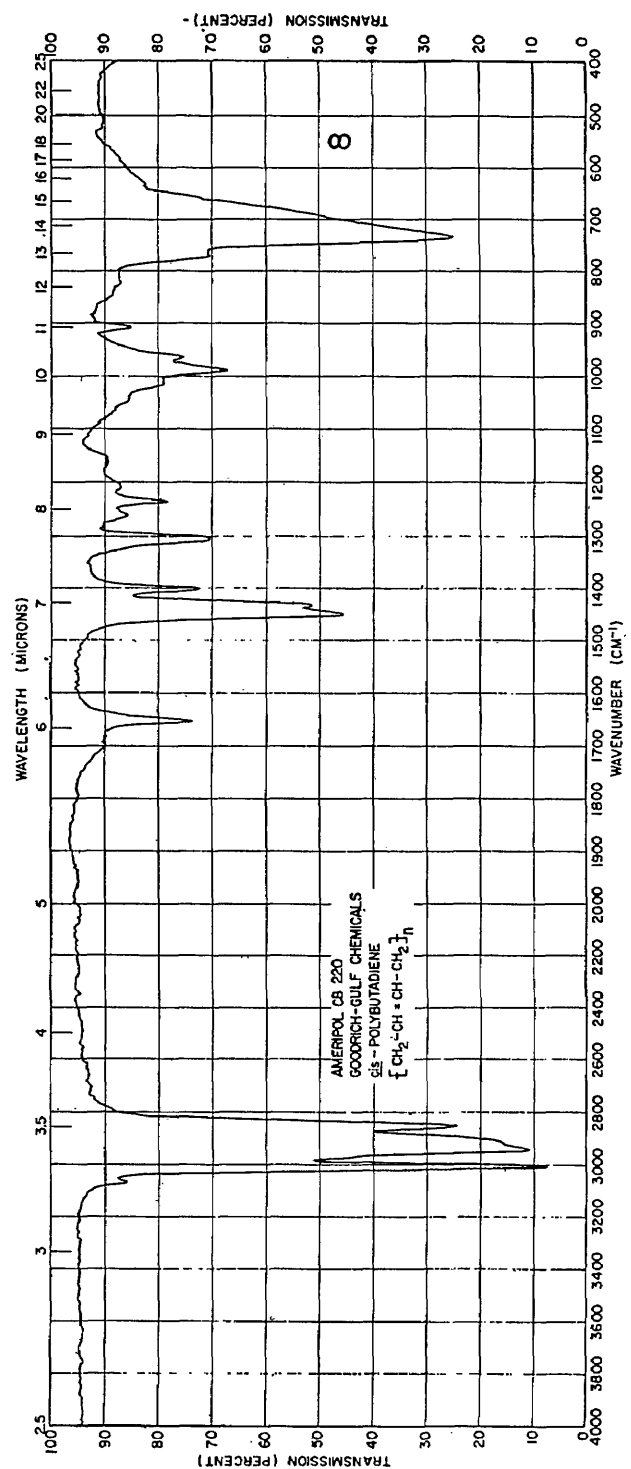
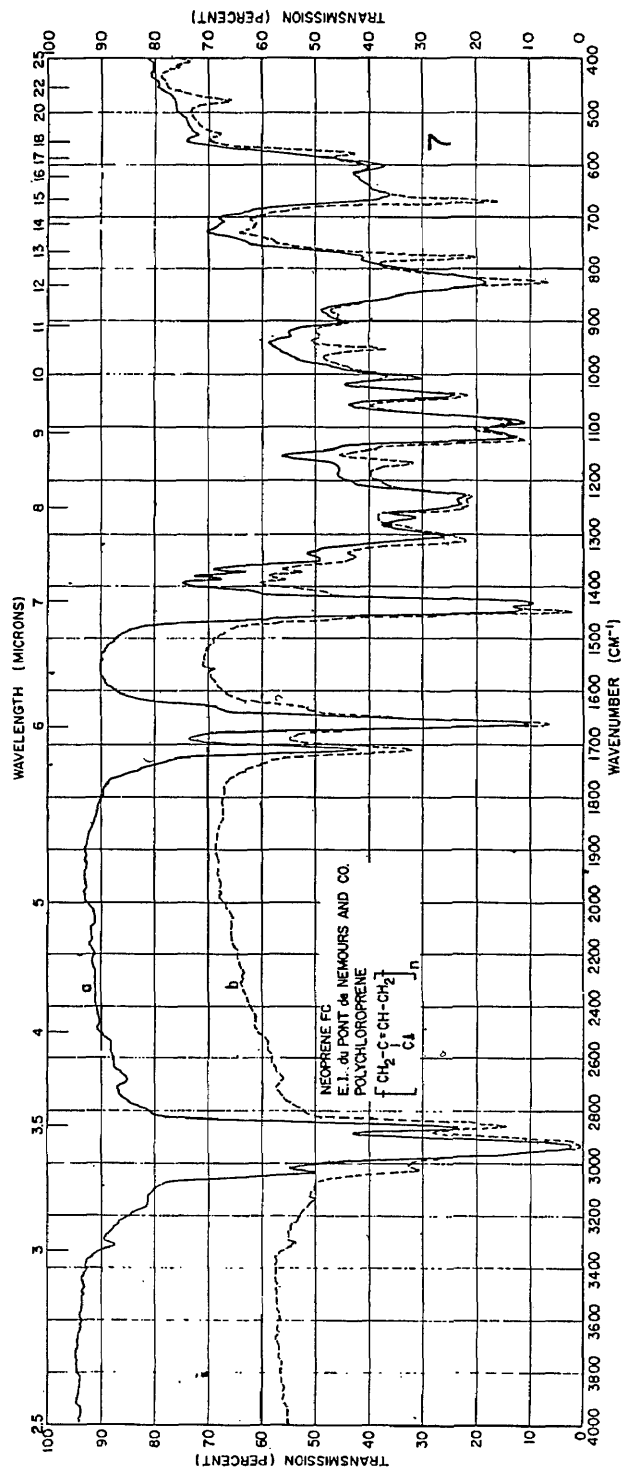
## SPECTRA OF ELASTOMERS

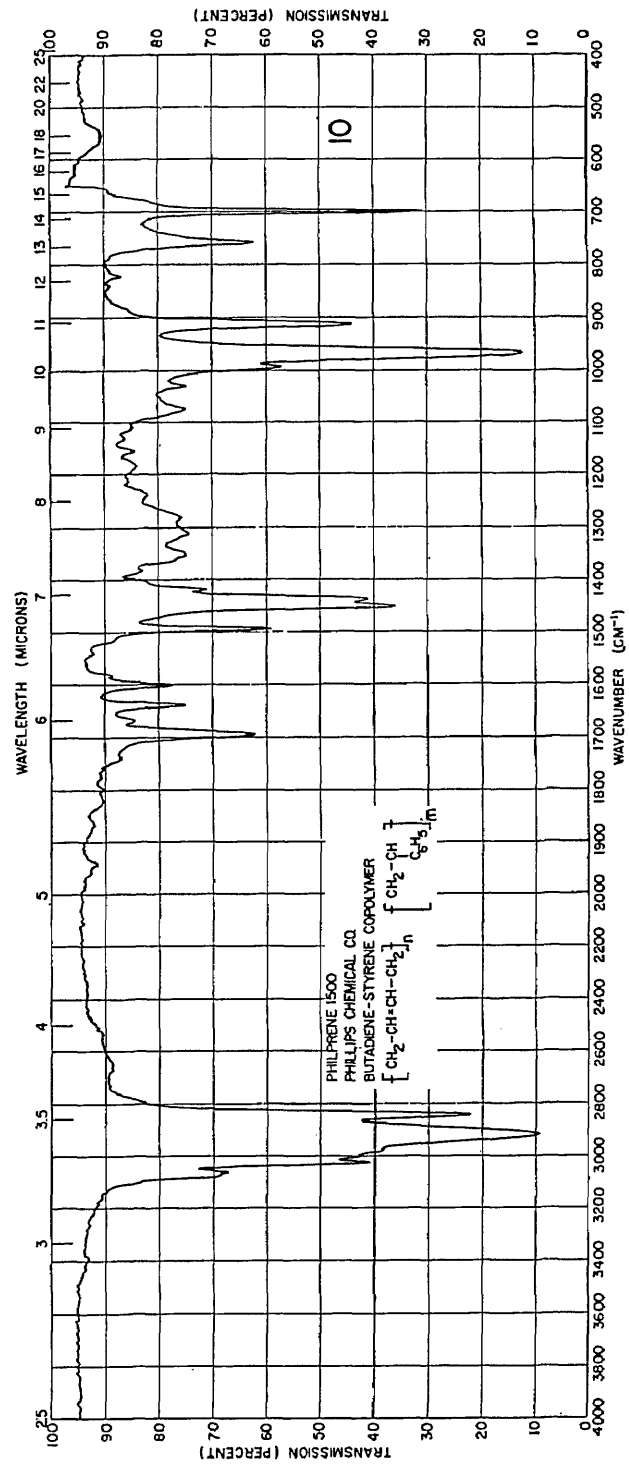
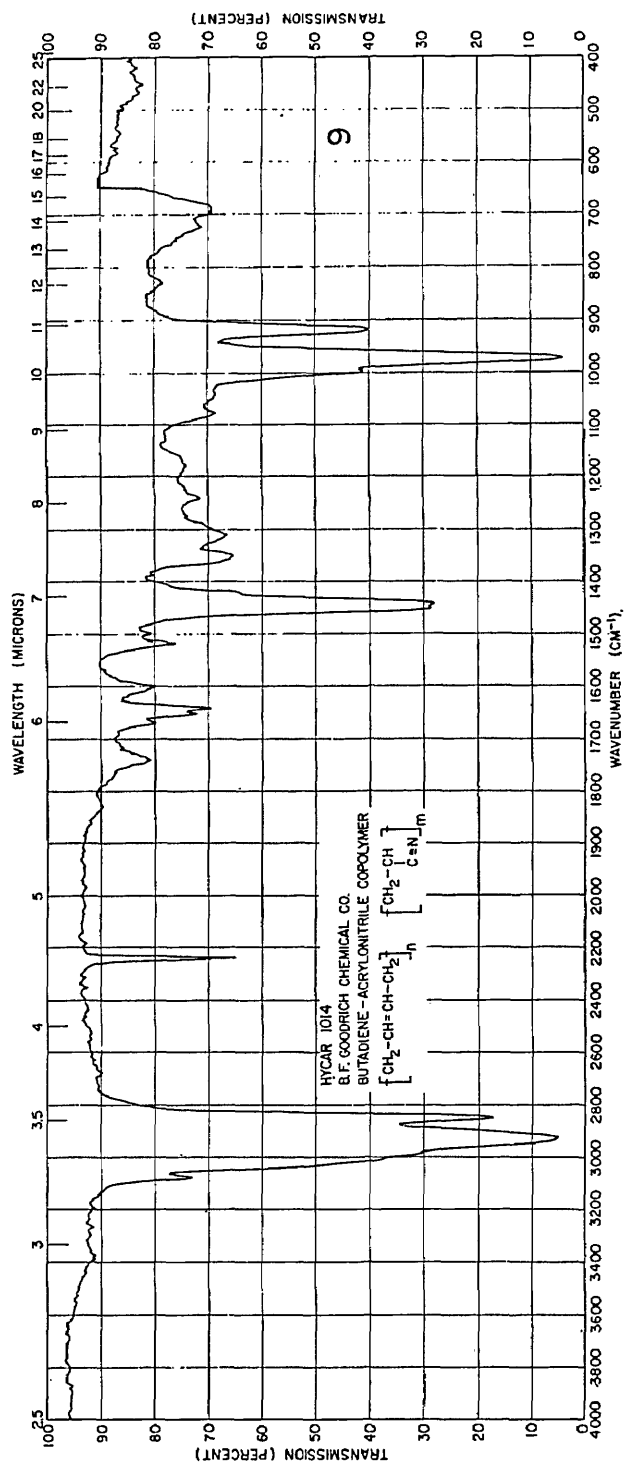


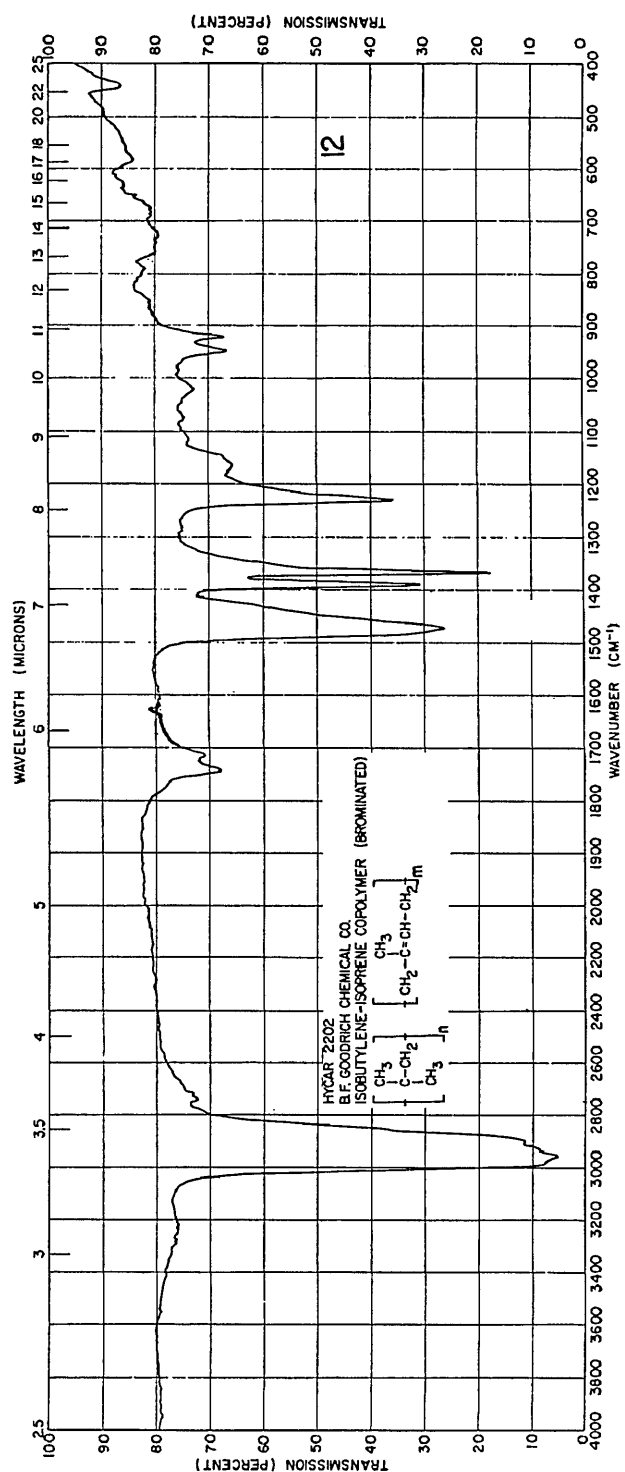
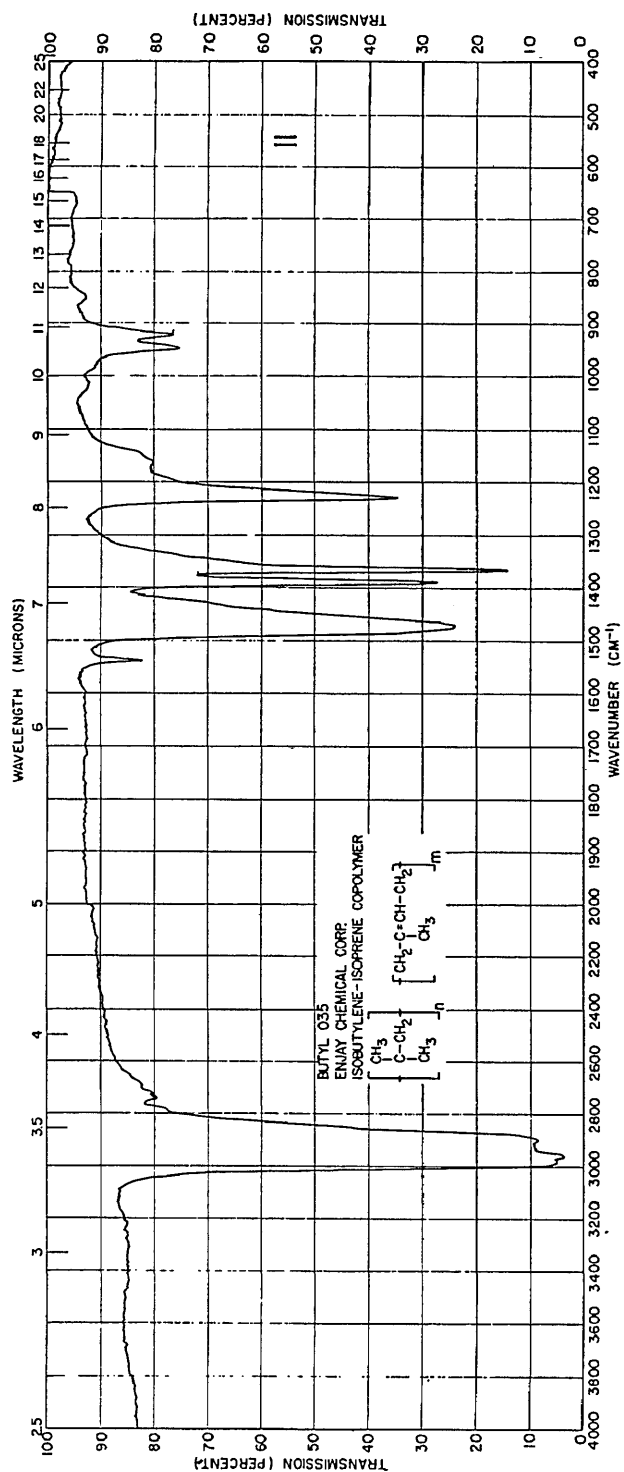


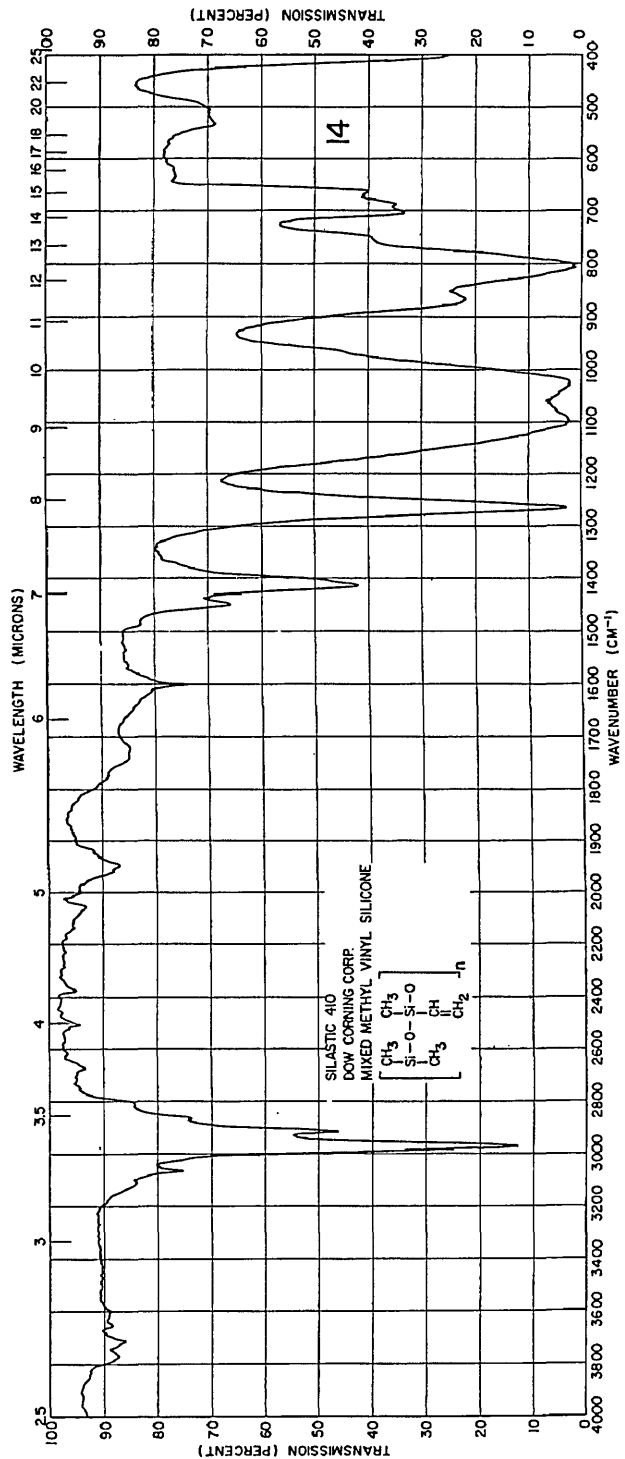
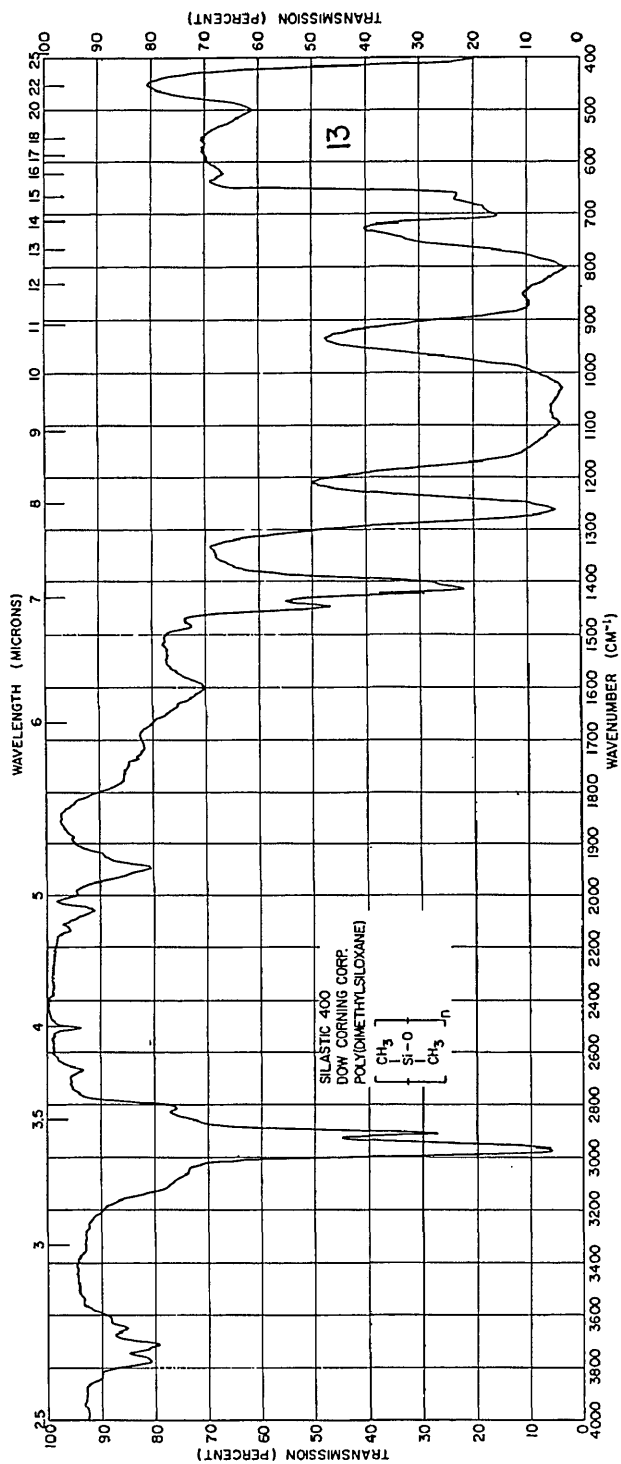


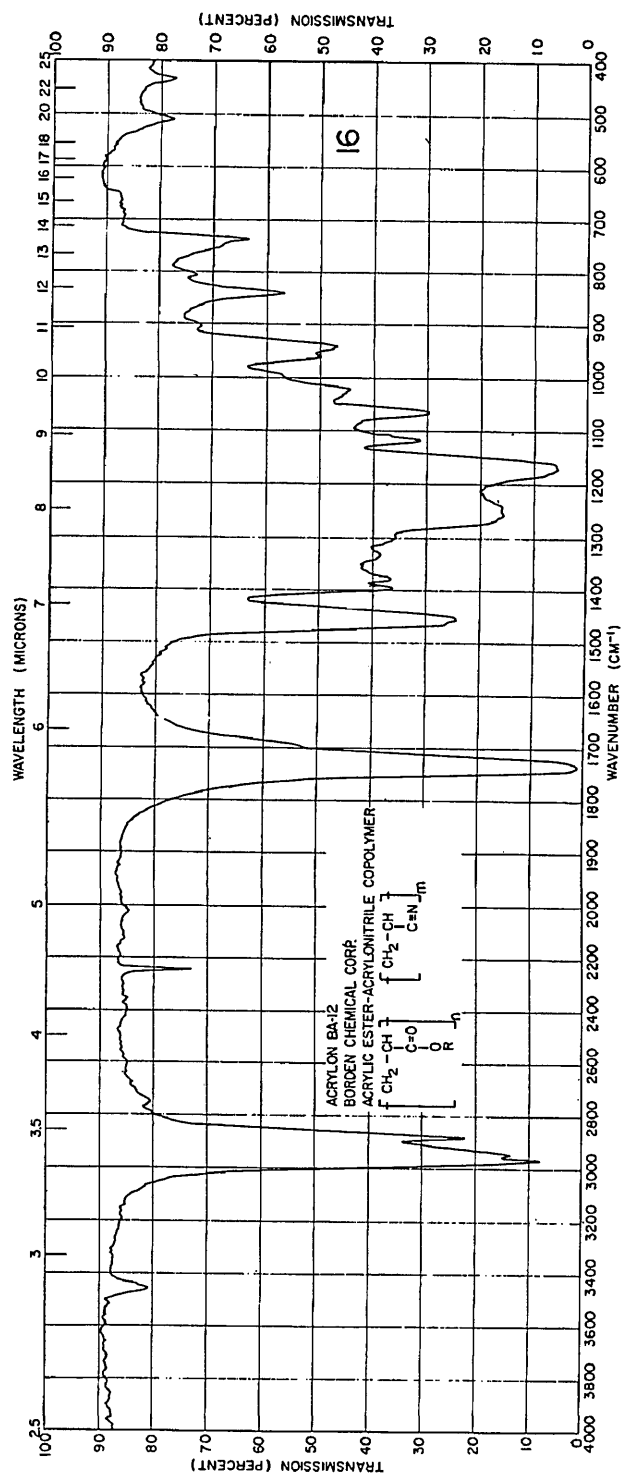


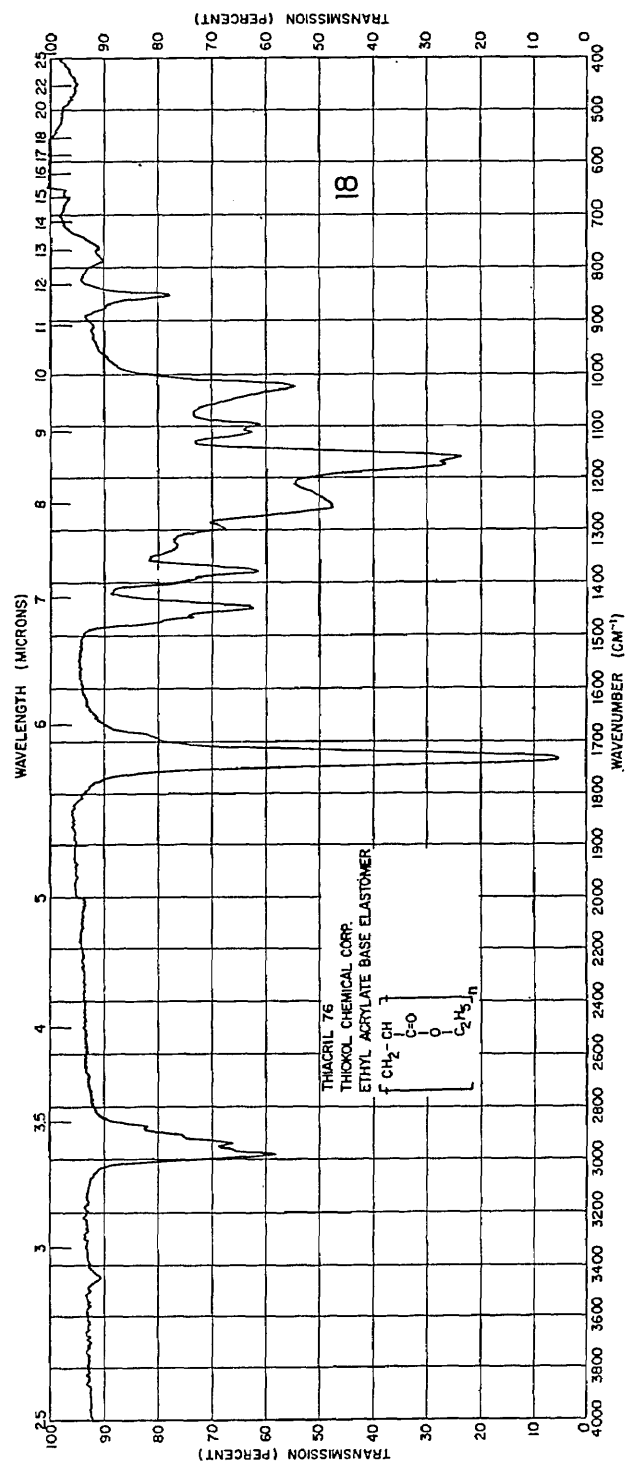
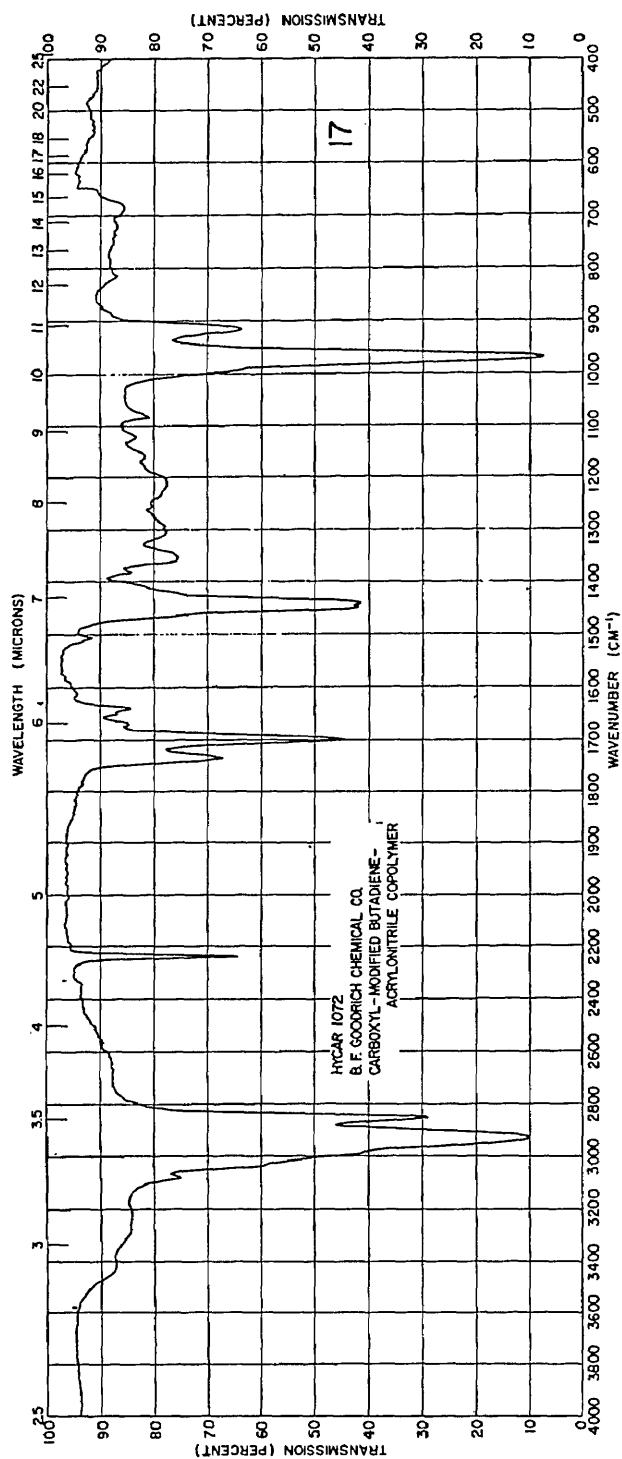




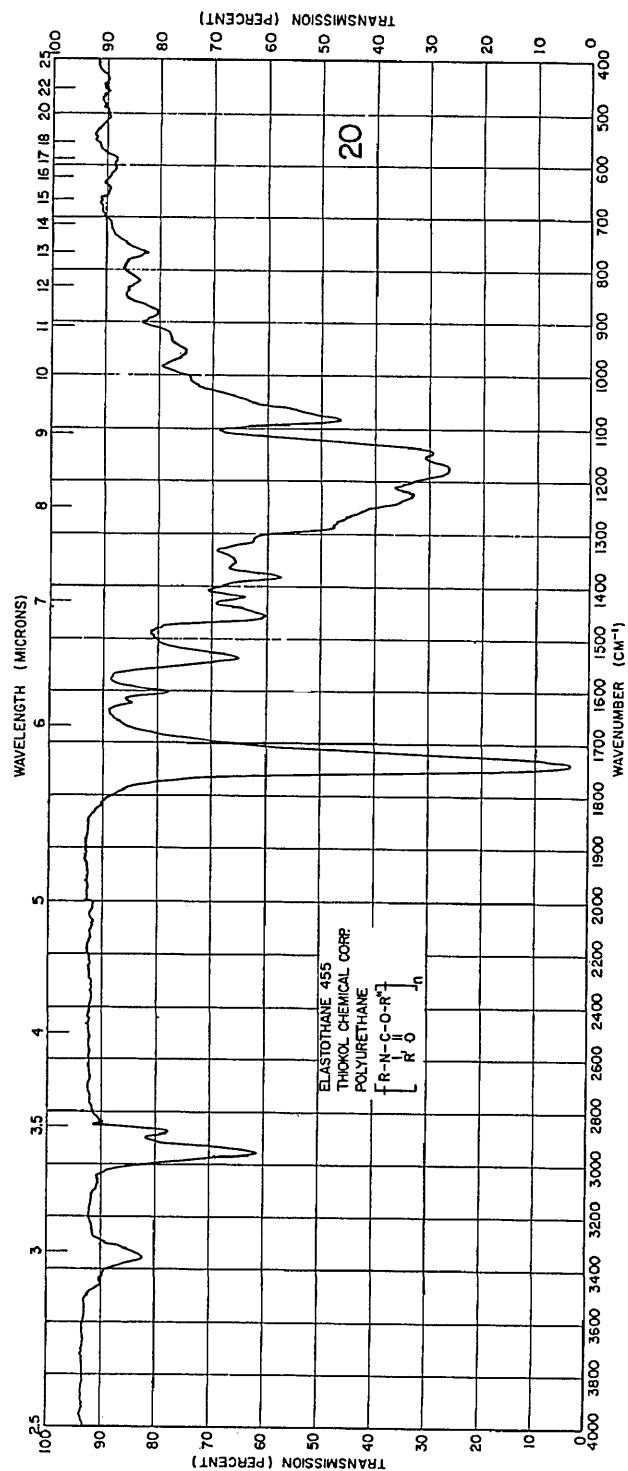
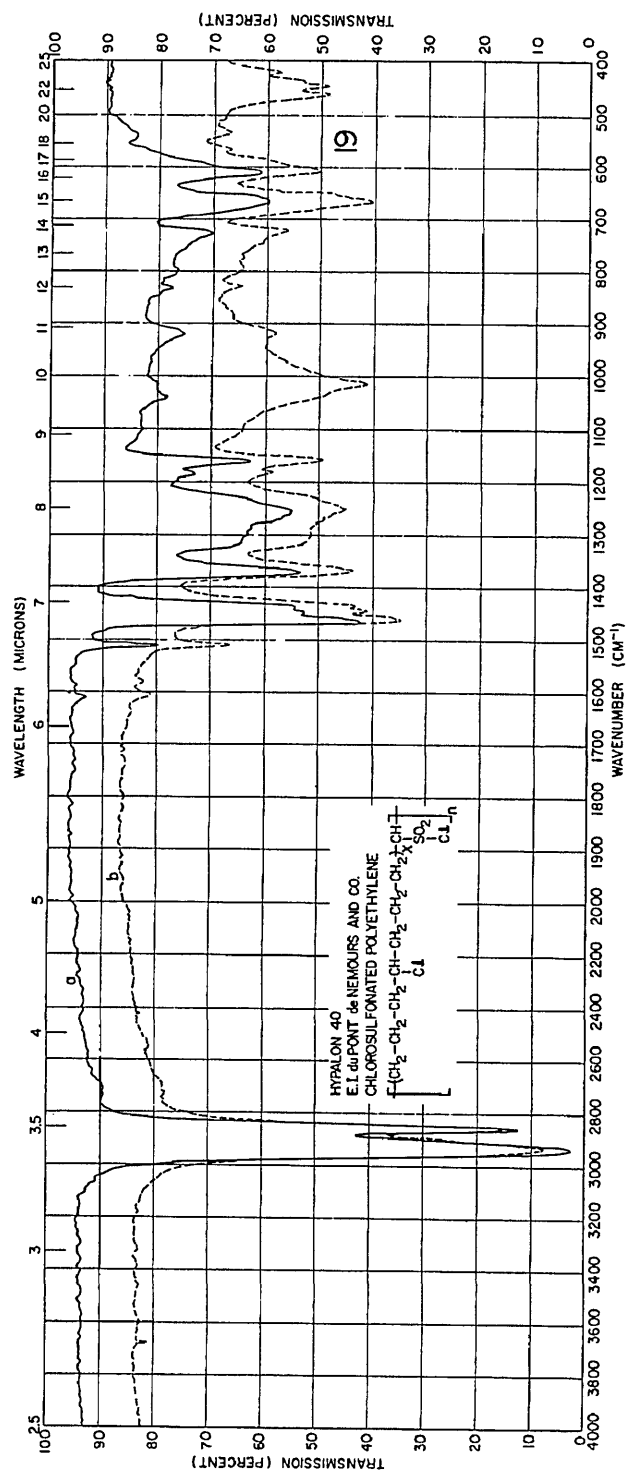


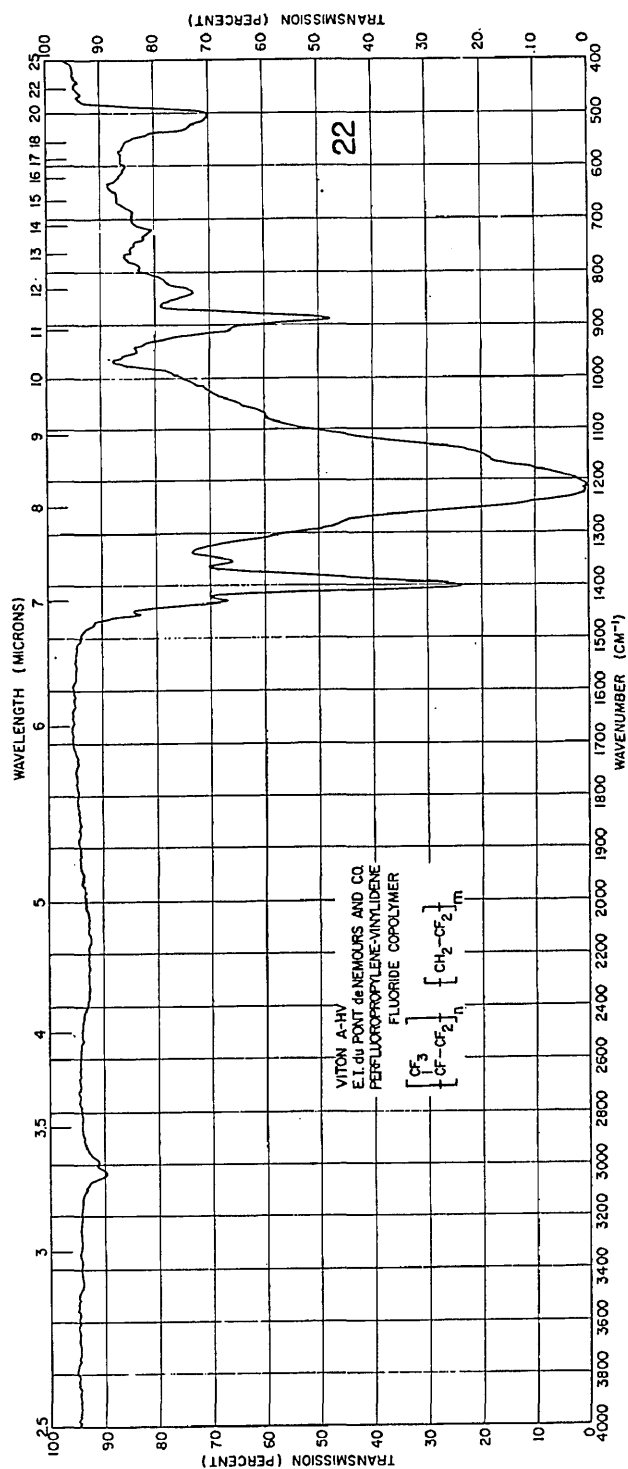
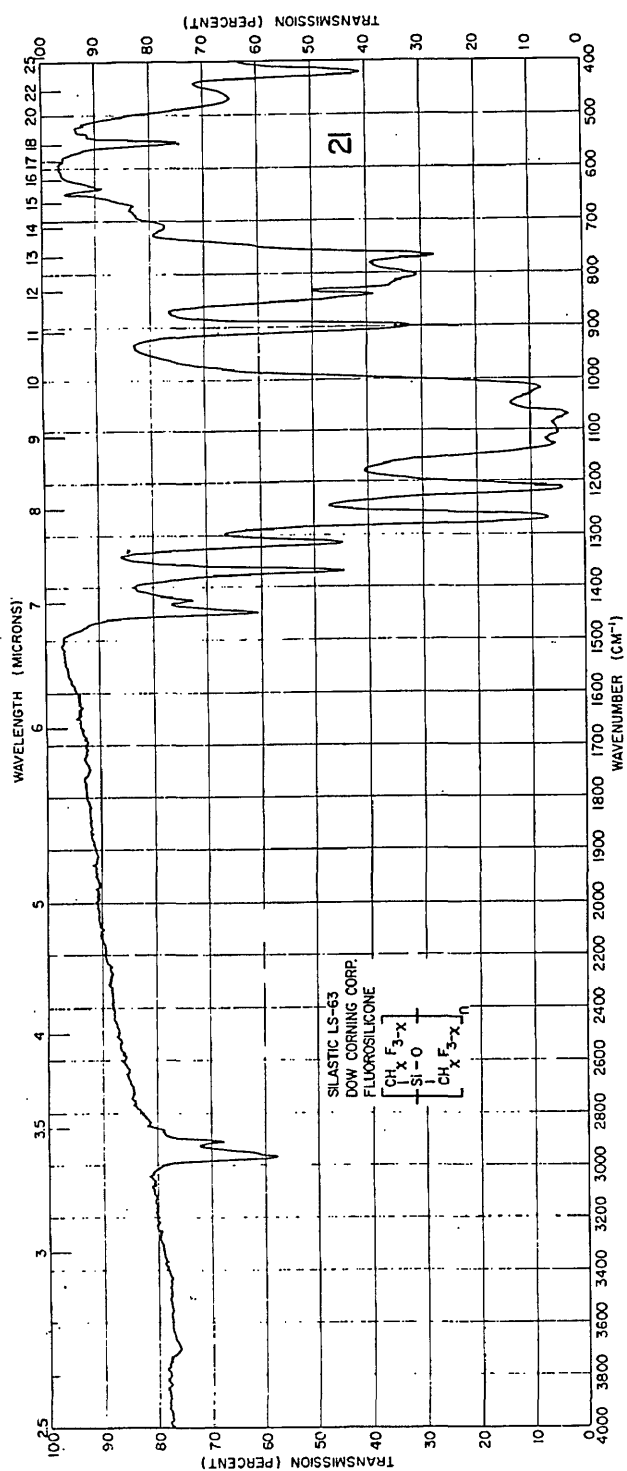


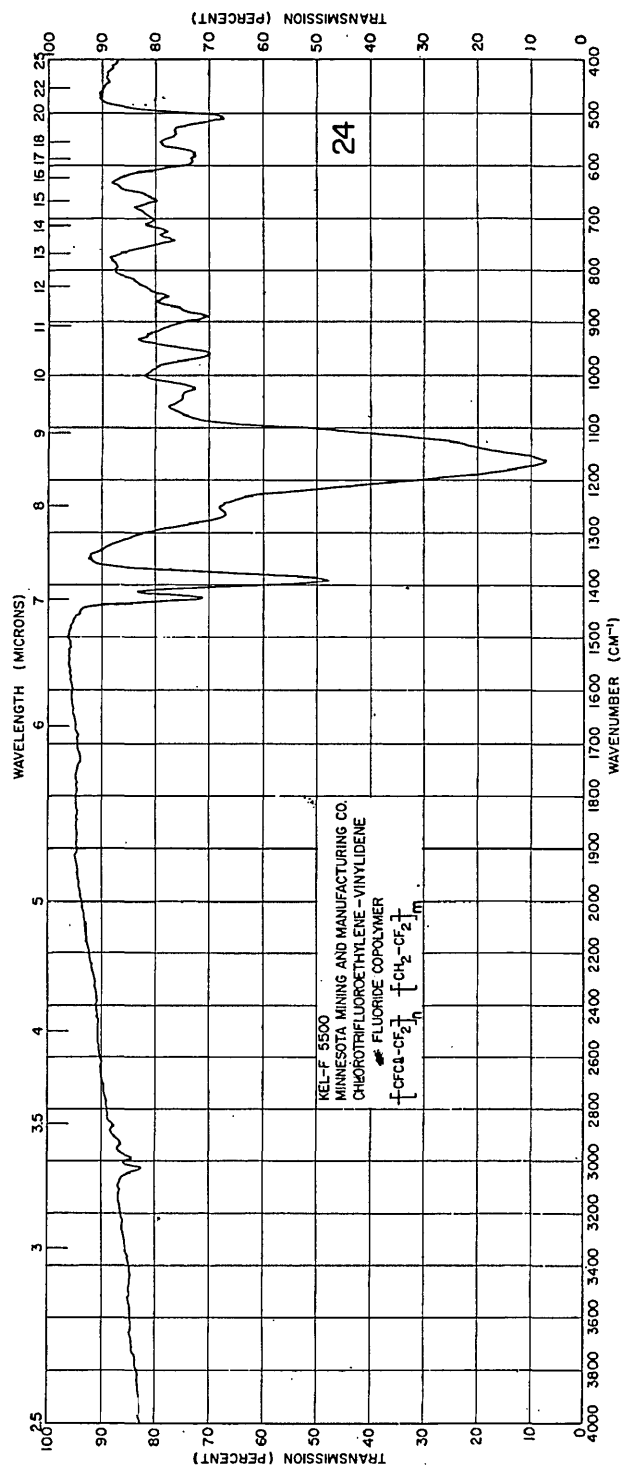
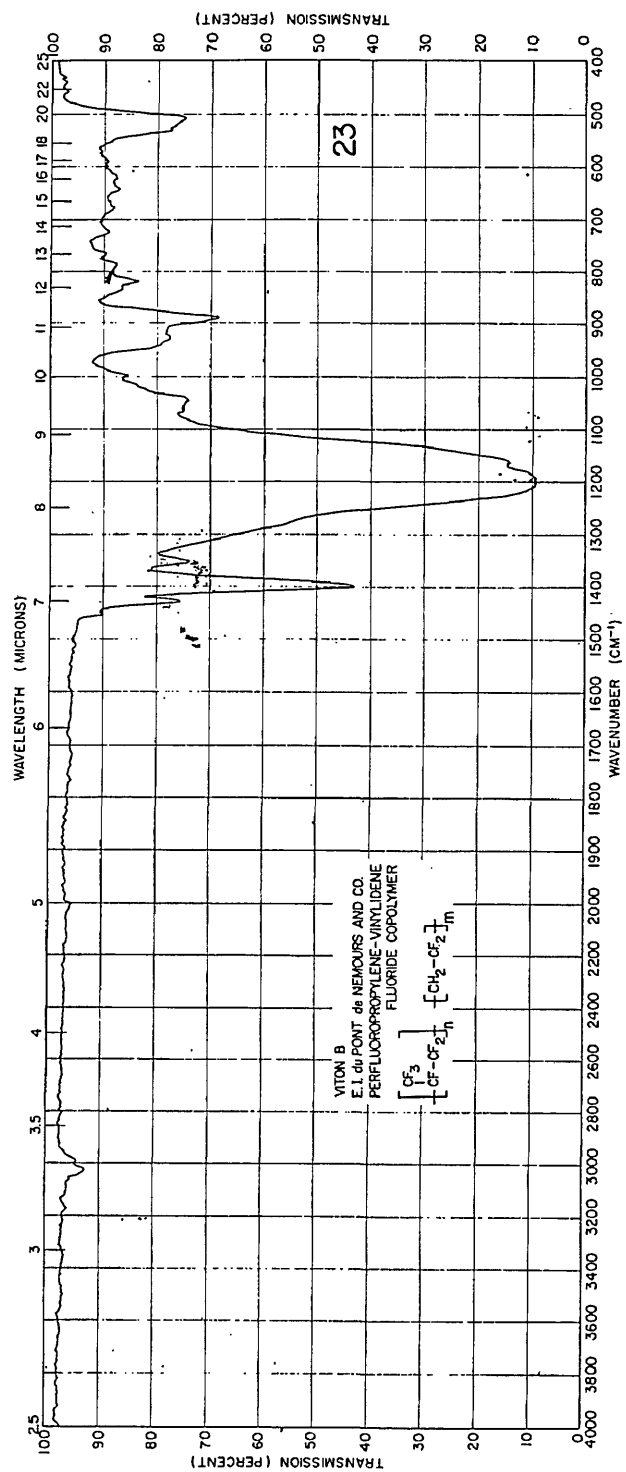


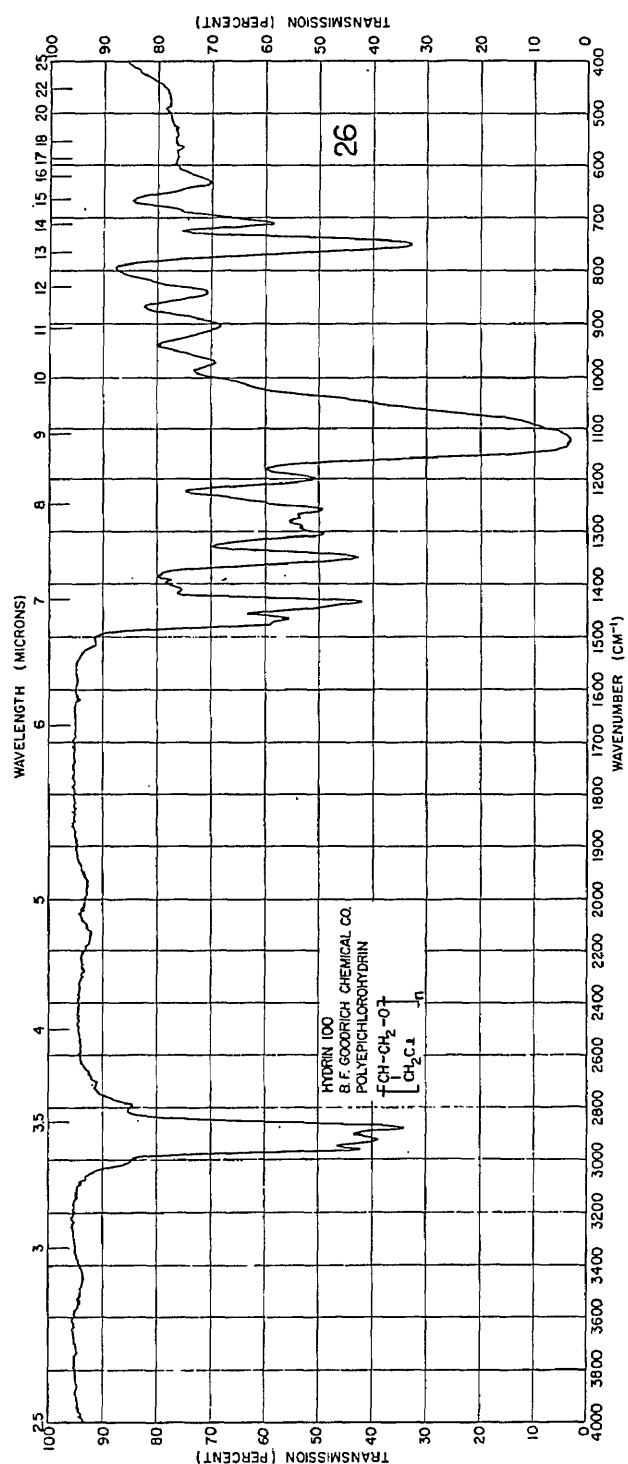
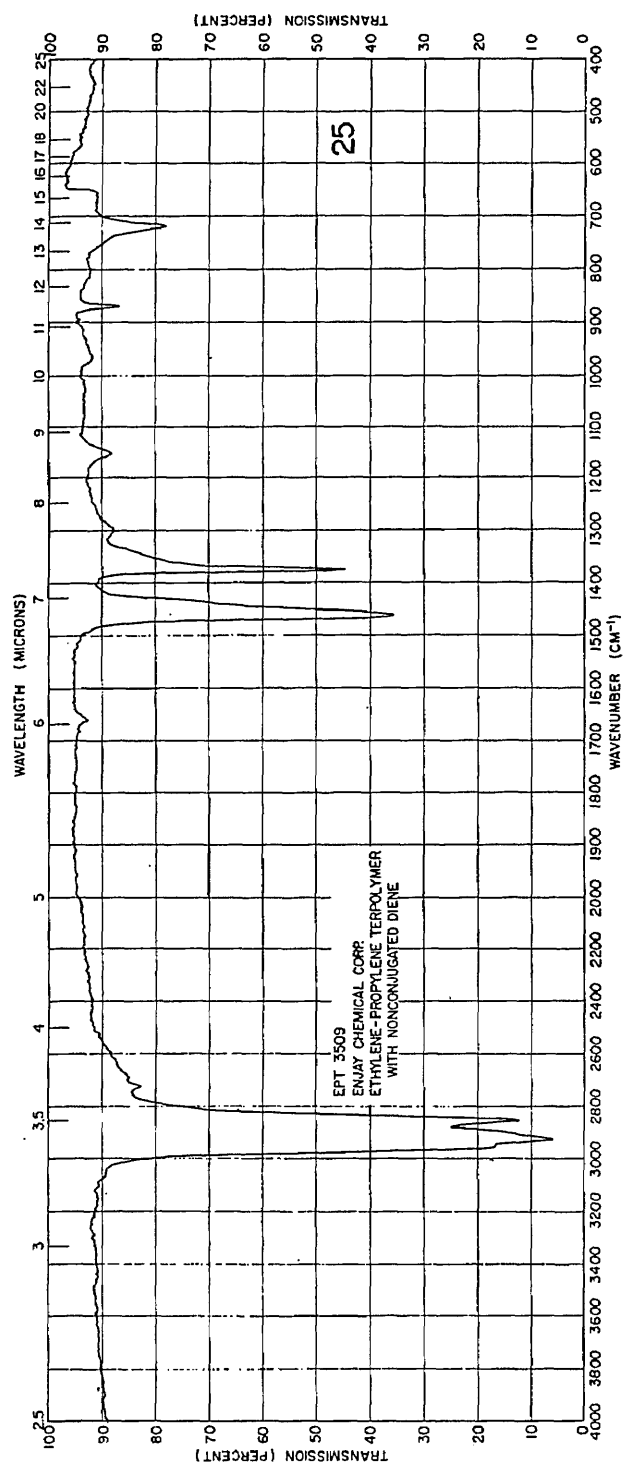


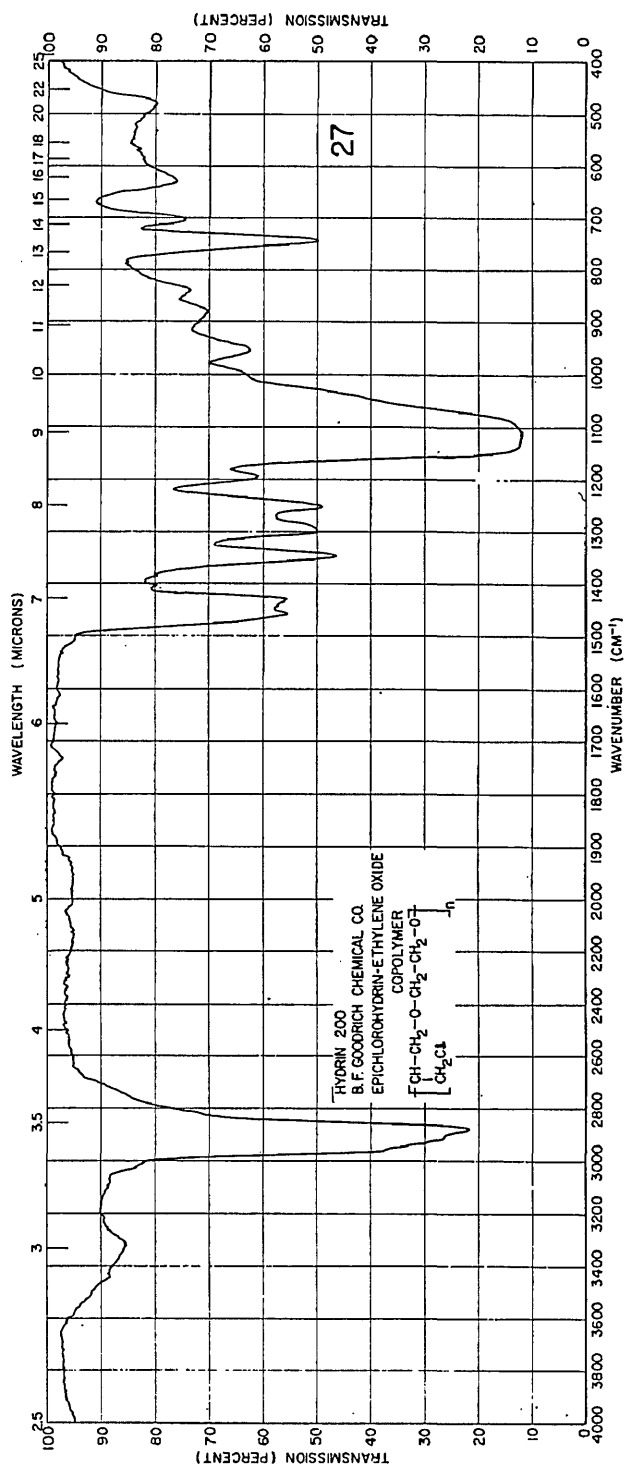












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13. ABSTRACT <p>Infrared spectra of thin films of 47 elastomers have been obtained in the region from 400 to 4000 <math>\text{cm}^{-1}</math>. The elastomers studied were uncured natural and synthetic rubbers, chosen as representative of the various classes of rubbers.</p> <p>The identification chart presented in Parts 1 and 2 of this series of reports has been expanded to include the elastomers studied here, in addition to the plastics used in that work. The use of this expanded chart and the reference spectra should make it possible to determine from the infrared spectrum of an unknown polymer its class or type.</p>			

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
SYNTHETIC RUBBER ELASTOMERS POLYMERS SPECTRA (INFRARED) IDENTIFICATION (RUBBER) ISOPRENE RUBBERS POLYSULFIDE RUBBERS CHLOROPRENE RUBBERS BUTADIENE RUBBERS ISOBUTYLENE-ISOPRENE RUBBERS SILICONE RUBBERS ACRYLIC RUBBERS CHLOROSULFONATED POLYETHYLENE URETHANE ELASTOMERS FLUORINATED RUBBERS ETHYLENE-PROPYLENE RUBBERS EPICHLOROHYDRIN ELASTOMERS REFERENCE SPECTRA						